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Anaerobic digestion of soft drink beverage waste and sewage sludge

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1 **Anaerobic digestion of soft drink beverage waste and sewage sludge**

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Abstract

Soft drink beverage waste (BW) was evaluated as a potential substrate for anaerobic co-digestion with sewage sludge to increase biogas production. Results from this study show that the increase in biogas production is proportional to the increase in organic loading rate (OLR) rate due to BW addition. The OLR increase of 86 and 171% corresponding to 10 and 20% BW by volume in the feed resulted in 89 and 191% increase in biogas production, respectively. Under a stable condition, anaerobic co-digestion with BW did not lead to any significant impact on digestate quality (in terms of COD removal and biosolids odour) and biogas composition. The results suggest that existing nutrients in sewage sludge can support an increase in OLR by about 2 kg COD/m³/d from a carbon rich substrate such as soft drink BW without inhibition or excessive impact on subsequent handling of the digestate.

Keywords: Anaerobic co-digestion, beverage waste, sewage sludge, biogas, organic loading rate.

1. Introduction

Anaerobic digestion is an integral component of municipal wastewater treatment, providing the efficient stabilisation and volume minimisation of sewage sludge through biological degradation (Wan et al., 2011, Sawatdeenarunat et al., 2016). In most wastewater treatment plants (WWTPs), sewage sludge is currently digested on its own. In recent years, concern about climate change and energy security has renewed the interest in anaerobic digestion as a platform for renewable energy production from organic wastes and sewage sludge (Berkessa et al., 2018, Tuyet et al., 2016, Li et al., 2017, Dennehy et al., 2018, Chu et al., 2015, Nguyen et al., 2017). Indeed, research activities in anaerobic co-digestion have gained significant momentum over the past decade (Luostarinen et al., 2009, Nghiem et al., 2017). Several water utilities around the world have begun to explore the possibility of co-digesting organic waste with sewage sludge using the spare digestion capacity at existing WWTPs (Tampio et al., 2016, Nghiem et al., 2017).

Anaerobic co-digestion involves the pairing of two or more organic wastes with complementary characteristics (Xie et al., 2016). In the context of a WWTP, sewage sludge is rich in nutrients (i.e. nitrogen and phosphorus) and contains all necessary micronutrients for the anaerobic process. On the other hand, organic wastes are a source of carbon for methane production but are often deficient in nutrients including micronutrients. Co-digestion can also benefit the anaerobic digestion process through the dilution of inhibitory substances that may originate from either sewage sludge or organic waste co-substrates (Mata-Alvarez et al., 2011). Furthermore, co-digestion presents an array of environmental and economic benefits. These include the diversion of putrescible wastes from landfill or incineration, the increase in the generation and feasibility of onsite renewable energy production and the added revenue offered through charging gate fees (Nghiem et al., 2017).

The selection for suitable co-substrate pairing and the optimisation of mixing ratios and organic loading rates (OLRs) are paramount to the widespread adoption of the practice. The prevailing substrate selection parameter for AD co-digestion in literature concerns the total organic carbon to total nitrogen (C/N) ratio of the feed solution, with the ideal ratio generally accepted to fall in the range of 15:1 and 30:1 (Weiland, 2010). Because substrates generally do not possess an ideal C/N ratio, co-digestion can mutually improve overall performance and stability. In general, sewage sludge has a low C/N ratio. Despite a somewhat limited biomethane potential, sewage sludge can provide a high buffering capacity and all the

necessary micronutrients for the anaerobic digestion process (Mata-Alvarez et al., 2014). Consequently, sewage sludge is arguably the most prevalent co-substrate in the current anaerobic digestion literature (Mata-Alvarez et al., 2011). In addition, due to the low carbon content in sewage sludge, the digesters at most WWTPs are operated at a low OLR.

The C/N ratio is not the only parameter that is important in regulating anaerobic co-digestion performance. Excessive inclusion of a carbon rich substrate (that can be rapidly hydrolysed into volatile fatty acids (VFAs), which are an intermediate product) can destabilise the anaerobic digestion process. Indeed, severe accumulation of these acids can cause the acidification of the reactor and subsequent inhibition of further digestion, i.e. methanogenesis. Furthermore, whilst it is attractive to utilise the spare digestion capacity in WWTPs for anaerobic co-digestion to enhance biogas production, there are some concerns regarding potential inhibition or negative implications for biosolids (solid fraction of digested sludge after dewatering) properties due to co-substrate addition. Thus the identification and demonstration of suitable co-substrates for sewage sludge co-digestion remain essential for the widespread uptake of the practice. Inhibition can lead to the reduction and even collapse of the biodegradation process. The primary cause of instability is the imbalance between methanogenic and acidogenic functional microbial groups, resulting from their variable requirements and growth kinetics (Chen et al., 2008). From an operational perspective, inhibition in co-digestion is seen to derive from both substrate selection and the mixing ratio adopted. These parameters can be represented through the OLR, which is subsequently the primary comparative measure for the operational ranges of different substrates.

The potential impact of co-digestion of sewage sludge upon biosolids quality and volume can restricts the implementation of the practice at full scale WWTPs. Biosolids management accounts for as much as 50% of the operational cost at some WWTPs (Semblante et al., 2014). Whilst in Europe biosolids are generally incinerated, in countries such as Australia and the USA, biosolids volume and quality are of higher importance as they are most commonly used for land application (Nghiem et al., 2017). In addition, some co-substrates may contain contaminants such as heavy metals and persistent organic chemicals. The occurrence of these contaminants in biosolids at a high concentration can render them unsuitable for land applications (Demirel et al., 2013, Bonetta et al., 2014). At the same time, there are a range of organic wastes in the urban environment that are both abundant and benign, making them highly attractive as a co-substrate for anaerobic co-digestion.

Beverage waste is a major source of organic substrate in metropolitan areas and a potential candidate for co-digestion with sewage sludge. It includes soft drink, alcoholic beverage, pre-mixed drink, and juice. Beverage waste such as soft drink consists primarily of water along with approximately 10-12% w/v dissolved carbon, mostly in the form of sugar (Isla et al., 2013). The volume of beverage waste produced annually is enormous. About 4.5 million m³/year of beverage waste is produced in Argentina. In the UK, it is estimated that 200,000 million m³ of beverage waste was produced in 2012 (Quested et al., 2013, Isla et al., 2013). Disposal of beverage production waste typically involves dilution into municipal wastewater streams, onsite treatment, or land spreading. **These all constitute a loss in the potential recoverable energy and may result in environmental pollution.** Despite the significant volume of beverage waste and its potential as a co-substrate for biogas production, the co-digestion of beverage waste has yet to be demonstrated in the current literature.

This work focuses on the optimisation of the co-digestion ratio between beverage waste and sewage sludge and the overall OLR in terms of biomethane production and system stability. The study further seeks to determine the likely type of inhibition associated with excessive concentrations of the co-substrate. Particular emphasis is directed toward elucidating the impact of co-digestion on the digestate quality in terms of biosolids odour potential.

2. Materials and Methods

2.1 Substrates

Anaerobically digested sludge was obtained from the Wollongong wastewater treatment plant (WWTP) in New South Wales (NSW) Australia and used as the inoculum. Primary sludge was obtained from the same plant every fortnight. A mixture of carbonated soft drinks was obtained from a commercial waste collector in NSW Australia. These soft drinks did not meet market requirements (e.g. out of date, damaged packaging, and contamination) and thus had to be destroyed and disposed. Diet and sugar free soft drinks were excluded from this study. The primary sludge and beverage waste were stored at 4 °C in the dark. Any unused portion of these substrates was discarded after two weeks of storage.

2.2 Experimental systems

Three identical anaerobic digesters were operated in parallel in this study. Each digester consisted of a 28 L stainless steel conical shape reactor, a peristaltic hose pump (DULCO[®] Flex from Prominent Fluid Controls, Australia), a biogas counter (Ritter CompanyTM,

MilliGascounter), a thermal probe and a gas trap for biogas sampling. A temperature control unit (Neslab RTE 7, Thermo Fisher Scientific, Newington, USA) was used to maintain the reactor temperature at 35 ± 1 °C. This was achieved by circulating hot water from the temperature control unit through a rubber tube that was firmly wrapped around the reactor. The reactor and pipeline were encased in polystyrene foam for insulation. The peristaltic hose pump was continuously operated to circulate the digestate at 60 L/h for mixing. Further details of these anaerobic digesters are available elsewhere (Yang et al., 2017).

2.3 Experimental Protocol

The working volume of each reactor was set at 20 L. At the beginning of this study, all three reactors were seeded with digestate from the Wollongong WWTP and were flushed with N₂ gas for 5 min. Unless otherwise stated, the hydraulic retention time (HRT) was set at 20 days. Each day, 1 L of digestate was removed from the digester and then 1 L of co-substrate (either primary sludge or a combination of primary sludge and beverage waste) was fed into the digester via the peristaltic pump.

Table 1: Operating conditions of the three anaerobic digesters over the 3 experiment stages.

		Reactor 1	Reactor 2	Reactor 3
Stage 1	OLR (kg COD/m ³ /d)	1.16	1.16	1.16
	Beverage waste (%)	0	0	0
	HRT (d)	20	20	20
	Duration (d)	52	52	52
Stage 2	OLR (kg COD/m ³ /d)	3.03	2.08	1.16
	Beverage waste (%)	20	10	0
	HRT (d)	20	20	20
	Duration (d)	31	31	31
Stage 3	OLR (kg COD/m ³ /d)	3.80	3.88	1.16
	Beverage waste (%)	30	20	0
	HRT (d)	20	15	20
	Duration (d)	25	25	25

The experiment was conducted over three stages (Table 1). In Stage 1, all three reactors were fed with primary sludge for 7 weeks to establish the baseline conditions. In the subsequent stages, Reactor 3 was used as the control system (same operating condition as in Stage 1) while Reactors 1 and 2 were used to evaluate the co-digestion of sewage sludge and beverage waste. In Stage 2, in addition to primary sludge, beverage waste was also fed into Reactors 1 and 2 at 20 and 10% (vol/vol) of the total feed, respectively. In Stage 3, the portion of beverage waste fed into Reactors 1 and 2 was increased further to 30 and 20% (vol/vol)

respectively. It is noted that the HRT of Reactor 2 was shortened to 15 d to achieve a similar OLR in both reactors.

2.4 Analytical Methods

TS, VS, alkalinity, total COD and soluble COD, pH and total organic acids (TOA) of the digestate were measured weekly. The primary sludge feed and beverage waste were also characterised on a weekly basis. COD measurements were conducted using a Hatch DRB200 COD Reactor and Hatch DR3900 spectrophotometer (program number 435 COD HR) following the US-EPA Standard Method 5220. Biogas composition analysis was conducted on a weekly basis by a portable gas analyser (GA5000 Gas Analyser, Geotechnical Instruments (UK) Ltd., England) using the gas trap to store the required 1 L gas sample prior to measurement. The details of these analytical techniques are available elsewhere (Yang et al., 2016, Nghiem et al., 2014).

Odour measurement was conducted based on an incubation technique previously reported by Glindemann et al. (2006). This method allows for the monitoring of hydrogen sulphite and six other sulphur bearing odour compounds. In brief, digestate was dewatered by laboratory centrifuge using the method previously developed by To et al. (2016). Then, 25 g of biosolids cake was collected into a 500 mL PET bottle. The bottle was sealed using a rubber cap and incubated at $28\pm1^{\circ}\text{C}$. The head space was extracted using a syringe at a specific time interval for Gas Chromatography – Mass Spectrometry analysis. The results are reported as volumetric concentration in the incubation bottle headspace.

3. Results and Discussion

3.1 Substrate Characteristics

Key properties of the inoculum, primary sludge and beverage waste are summarised in Table 2. Beverage waste contained a significantly higher organic fraction mostly in the form of dissolved sugars than the primary sludge. On the other hand, VS of the beverage waste is only marginally higher than that of primary sludge (Table 2). Given the high carbon content and easily degradable organics (owing to the high sugar content) of beverage waste, its co-digestion with nutrient rich, high buffering capacity primary sludge can provide complementary benefits to both co-substrates. Co-digestion is necessary to achieve a balance between organic carbon and nutrients to prevent pH-derived inhibition based on the rapid formation of intermediate products, specifically volatile fatty acids.

Table 2: Characteristics of the inoculum, primary sludge feed, and beverage waste (mean \pm standard deviation of at least 5 samples).

	Inoculum	Primary sludge	Beverage waste
TS (%) (fresh weight)	0.85	1.7 \pm 0.2	4.73 \pm 0.5
VS (%) (fresh weight)	0.56	1.3 \pm 0.1	4.59 \pm 0.5
VS/TS (%)	65	78 \pm 1	92 \pm 1
pH	6.6	6.1 \pm 0.5	3.3 \pm 0.1
COD (mg/L fresh weight)	8300	22300 \pm 1750	204000 \pm 27000

3.2 Process Performance

3.2.1 Biomethane Production

In Stage 1, the three reactors were preconditioned under the same operating conditions. As expected, biomethane production from all three reactors was almost identical during this stage (Figure 1 A). In Stage 2, stable biomethane production was observed during the co-digestion of sewage sludge with beverage waste co-digestion ratios of 10 and 20%, corresponding to an increase in OLR of 86 and 171% and in overall biogas production of 89 and 191% (compared to the control reactor).

During the early phase of Stage 2, an elevated methane content of approximately 70% was observed in the biogas produced from Reactors 1 and 2 compared to the baseline value of 60% from Reactor 3 (Figure 1 B). However, at the end of Stage 2, the methane content in biogas from Reactors 1 and 2 returned to the baseline. The brevity of this increase in methane content suggests the change may result from the transitory condition of co-digestion with carbohydrate rich co-substrates. A permanent increase in methane content was observed by Jang et al. (2016) during the co-digestion of food waste and waste activated sludge, corresponding to higher food waste mixing ratios. However, it is noted that the initial methane content recorded in previous feeding conditions in Jang et al. (2016) was usually low (i.e. only 50%) and their observations concern the co-digestion of different co-substrates. The disparity between our findings and that from Jang et al. (2016) highlights the need for further research to ascertain the potential effect of co-digestion on methane content.

In addition to the overall biomethane production, co-digestion may also affect the specific methane yield. Over the duration of the experiment the methane yield of the sewage sludge was 284 mL CH₄/g COD added, suggesting a highly degradable sludge substrate compared to typical values in literature, often in the range of 188 to 214 mL CH₄/g COD (Astals et al., 2013). It is noted that in Stage 2, the specific methane yield of the control (Reactor 3)

deceased slightly from 300 to 275 mL/g COD added. On the other hand, the specific methane yield of Reactors 1 and 2 were stable despite the addition of beverage waste (Figure 2). This is consistent with the findings by Razaviarani et al. (2013), who observed a slight increase in the specific methane yield when co-digesting sewage sludge with glycerine. Further results suggest the increase was likely due to the higher digestible fraction of beverage waste relative to sewage sludge, rather than due to any synergistic effect.

Inhibition of both co-digestion reactors was observed during Stage 3 at an OLR of ~3.8-3.9 kg COD/m³/d. During Stage 3, both the volume and methane content of the biogas declined sharply in Reactors 1 and 2. When applying mono-digestion of a similar soft-drink wastewater, Redzwan and Banks (2007) observed complete inhibition of methanogenic processes at a loading rate of 1.33 kg COD/m³ in batch experiments, which was attributed to alkalinity loss (from 2300 mg/L to 1000 mg/L) and accumulation of volatile fatty acids, which reached a concentration of 1500 mg/L. Whilst this inhibitory OLR value was obtained in batch experiments and therefore is not directly comparable, it demonstrates the risk of overloading when co-digesting with organic rich co-substrates. Furthermore, the stable operation demonstrated at a much higher OLR in this study suggests that co-digestion of the substrate with sewage sludge can improve digestion stability.

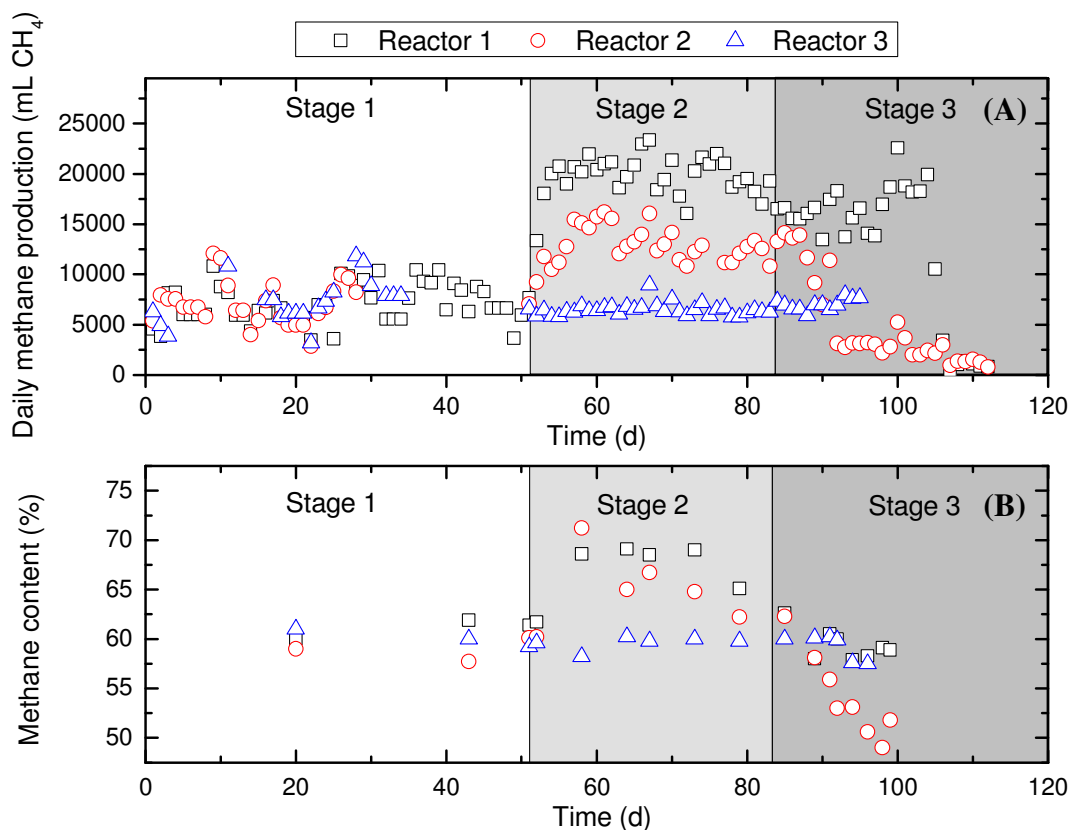


Figure 1: Biomethane Production: (A) Daily biomethane production at different experimental stages and (B) Methane content in biogas during different experimental stages (temperature = 35 ± 1 °C; other experimental conditions are as described in Table 1).

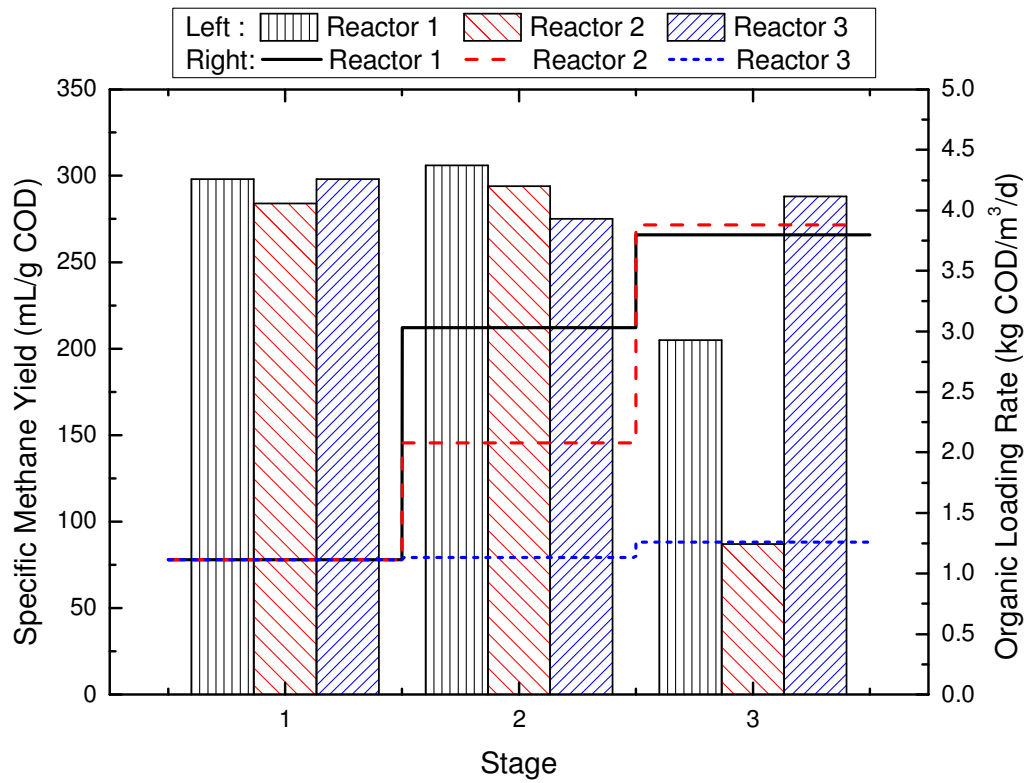


Figure 2: Specific methane yields at each OLR value (temperature = 35 ± 1 °C; other experimental conditions are as described in Table 1).

3.2.2 Digestate Quality

The removal efficiency of both VS and COD was constant throughout Stages 1 and 2 of the experiment, indicating that co-digestion does not significantly impact on the performance of anaerobic digestion (Figure 3). Nevertheless, a small increase in VS and COD in the digestate from Reactors 1 and 2 during Stage 2 could be observed. At steady-state condition in Stage 2, the average VS content in the digestate from Reactor 1 and 2 by 21% and 27% respectively, compared to Stage 1. Similarly, the average total COD in the digestate from Reactor 1 and 2 also increased by 18% and 23% compared to Stage 1. This is a small increase compared to the increase in OLR of 171 and 86% in Reactor 1 and 2, respectively. Indeed, this small

increase in VS and COD in the digestate is expected and can be attributed to a high OLR value. During Stage 2 the ratio of VS/TS in the digestate was relatively constant in Reactors 1, 2 and 3, with ratios of 63, 65 and 62% respectively.

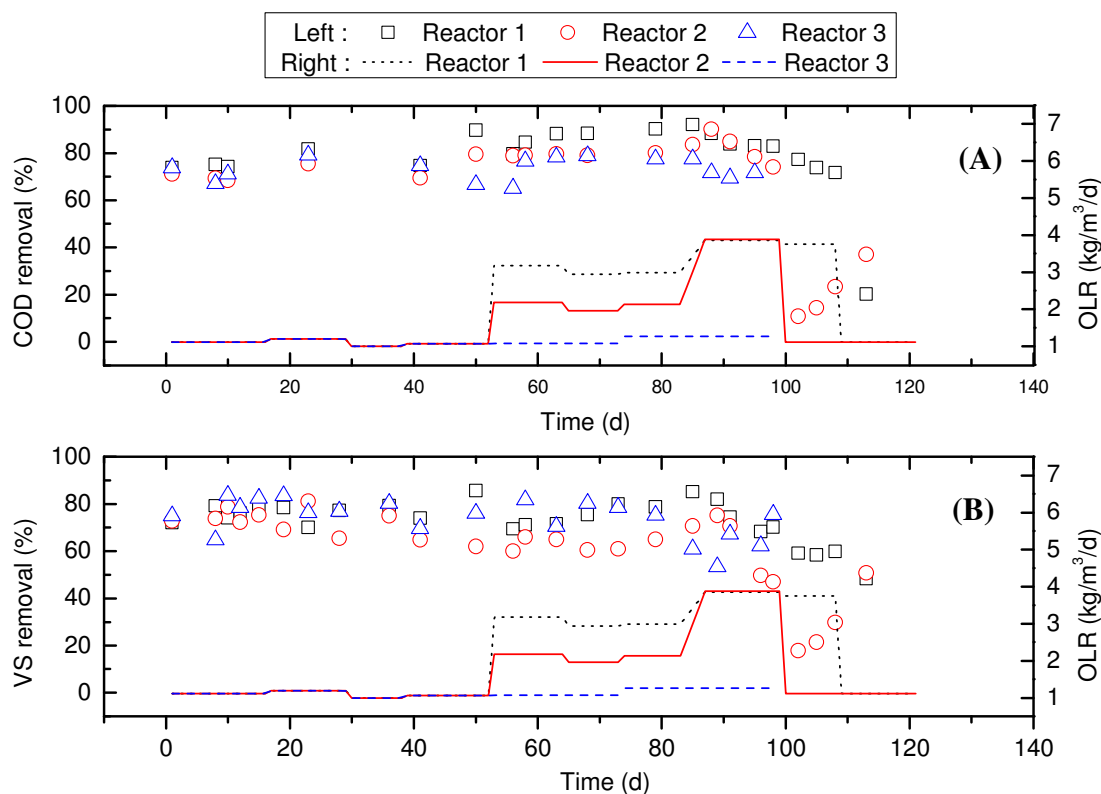


Figure 3: Biosolids stabilisation: (A) Volatile removal efficiency and (B) total COD removal efficiency with corresponding OLR value (temperature = 35 ± 1 °C; other experimental conditions are as described in Table 1).

Soft drinks only contain sugar and flavours, thus as expected, their co-digestion with sewage sludge does not result in any notable impact on biosolids odour. Similar to the VS and COD removal data, under a stable condition (Stage 2), the odour potential of biosolids samples from all three Reactors was almost identical (Figure 4). It is also noteworthy that among the seven sulphur bearing odour compounds (namely H_2S , CH_4S , C_2H_6S , $C_2H_6S_2$, CS_2 , $(CH_3)_2S$, COS) from biosolids monitored in this study, only hydrogen sulfide (H_2S) was prevalent in all samples. In most instances, the concentration of H_2S in the head space of biosolids samples from Reactor 3 (mono digestion) was slightly higher than that from Reactor 1 and 2 (co-digestion).

3.2.3 Impact of co-digestion on specific methane yield

The co-digestion of beverage waste with primary sludge was evaluated using a COD balance approach, previously adopted in Aichinger et al. (2015). A COD balance was used to represent the digestion performance of each of the reactors during Stage 2 (Figure 5). The specific biomethane yields and subsequent COD consumption were determined for the mono-digestion of each substrate. Reactor 3 demonstrated a biomethane yield of 275 mL/g COD added, which was used to represent sewage sludge COD consumption. Meanwhile a yield of 321 mL/g COD was adopted for beverage waste, derived from previous biomethane potential evaluation of the substrate (Wickham et al., 2016). Based on this data, the conversion of COD into biomethane in each substrate precisely matched their performance during mono-digestion. Results in Figure 6 show that beverage waste was fully digested. In other words, beverage waste addition did not result in any discernible increase in the COD content of the final digestate. On the other hand, data in Figure 5 cannot be used to confirm the synergistic effect of sewage sludge and beverage waste co-digestion. The lack of observable synergism is not unexpected as substrates rich in rapidly degradable organic matter have been observed to produce little to no synergetic effects during co-digestion (Jensen et al., 2014).

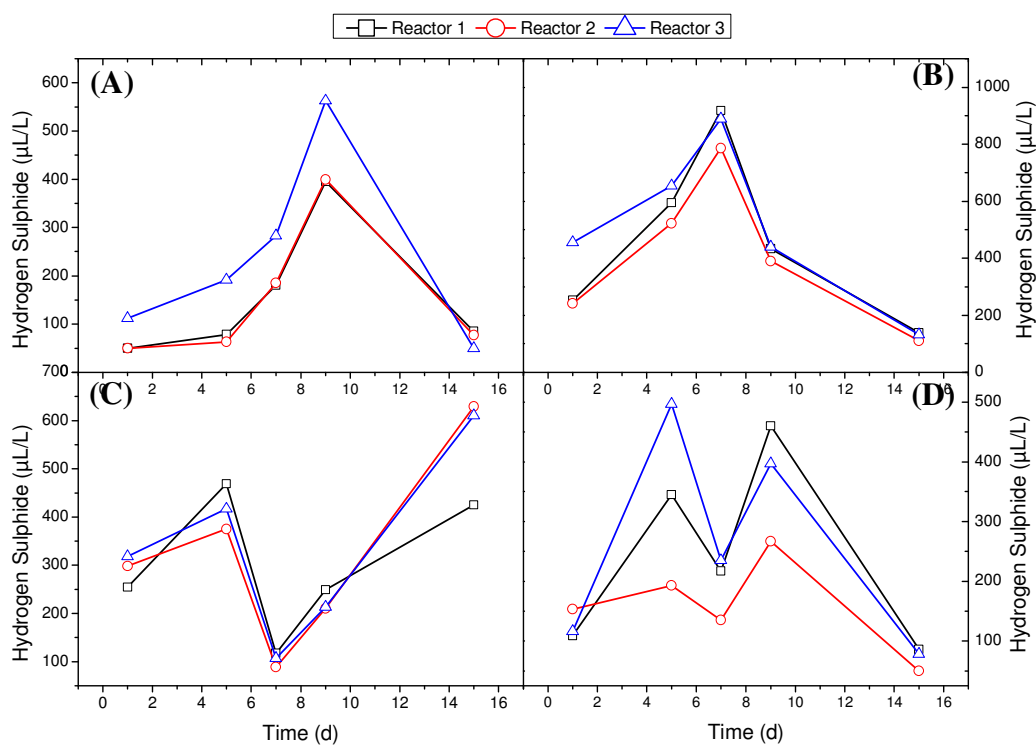


Figure 4: Odour generation over 15 days from biosolids produced during Stage 2, sampled on (A) day 64, (B) day 71 (C), day 78 and (D) day 85 (Reactor 1: co-digestion with 20% (v/v) beverage waste, Reactor 2: co-digestion with 10% (v/v) beverage waste and Reactor 3: mono-digestion of primary sludge) temperature = 35 ± 1 °C; other experimental conditions are as described in Table 1.

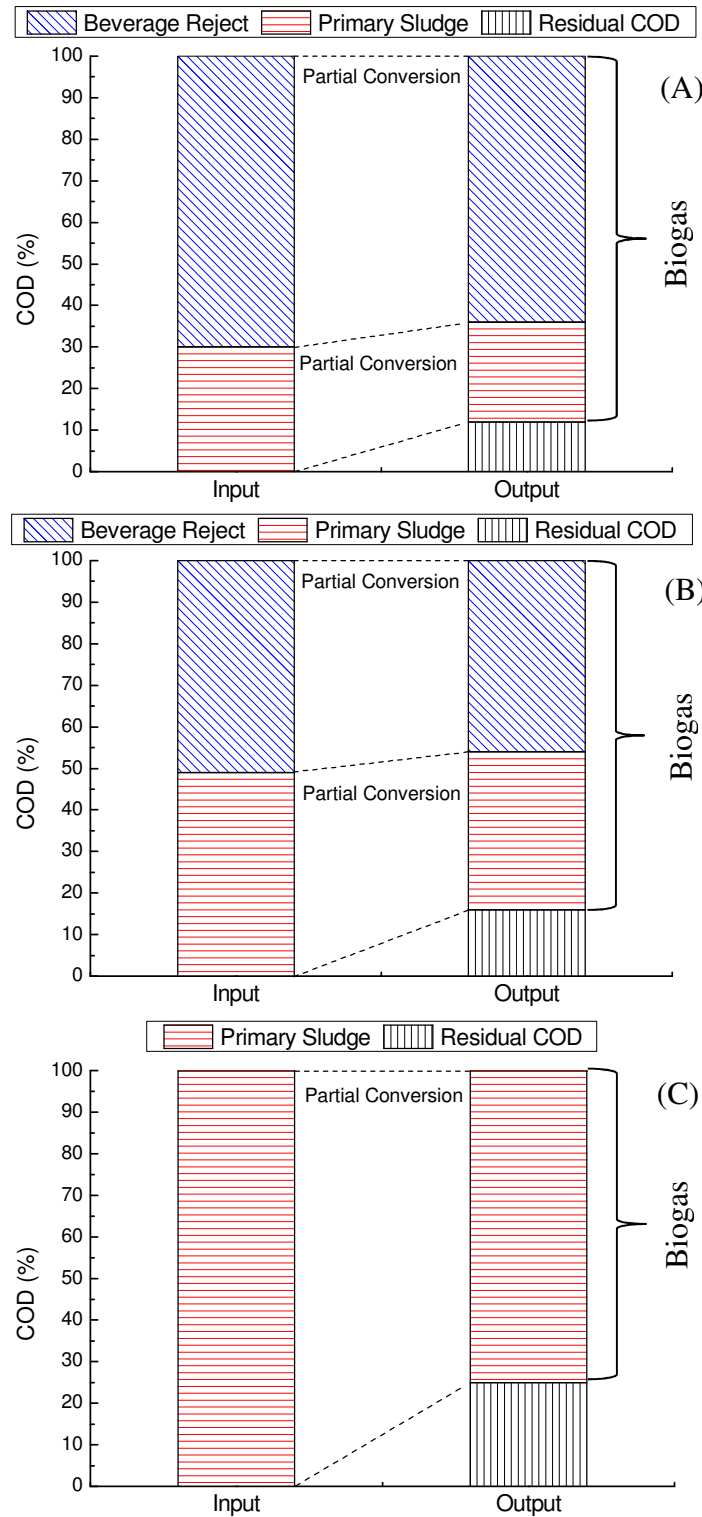


Figure 5: Average substrate mixture and corresponding gas production represented as COD balance during Stage 2 for (A) Reactor 1 (co-digestion with 20% (v/v) beverage waste), (B)

Reactor 2 (co-digestion with 10% (v/v) beverage waste) and (C) Reactor 2 (mono-digestion of primary sludge) Temperature = 35 ± 1 °C; other experimental conditions are as described in Table 1.

3.3 Process Stability

Process stability was evaluated through the measurement of soluble COD, alkalinity, TOA, and pH. Stable co-digestion was observed in each of the parameters throughout Stages 1 and 2 (Figure 6). A slight decline in alkalinity in Reactors 1 and 2 relative to the control was observed. However; this was not accompanied by any accumulation of TOAs or significant drop in pH, indicating the degradation of BW generates less alkalinity than that of sewage sludge. A sharp decline in the stability of Reactors 1 and 2 occurred with the commencement of Stage 3. The higher OLR values adopted in these reactors instigated the rapid accumulation of COD in the form of organic acids, leading to the consumption of alkalinity and sharp decline in pH. Indeed, in Stage 3, the profiles of soluble COD and TOA in Reactors 1 and 2 closely resemble each other (Figure 6). The progression of inhibition aligns well with previous findings, as carbohydrate rich co-substrates are known to pose risks in the accumulation of intermediaries such as volatile fatty acids (Astals et al., 2014). It is noteworthy that a similar rate of inhibition was observed in both in Reactors 1 and 2. In Stage 3, these reactors have similar OLR value but different HRT (20 vs 15 days). These results suggest that inhibition was intrinsically due to a high OLR value rather than the sudden variation in organic loading.

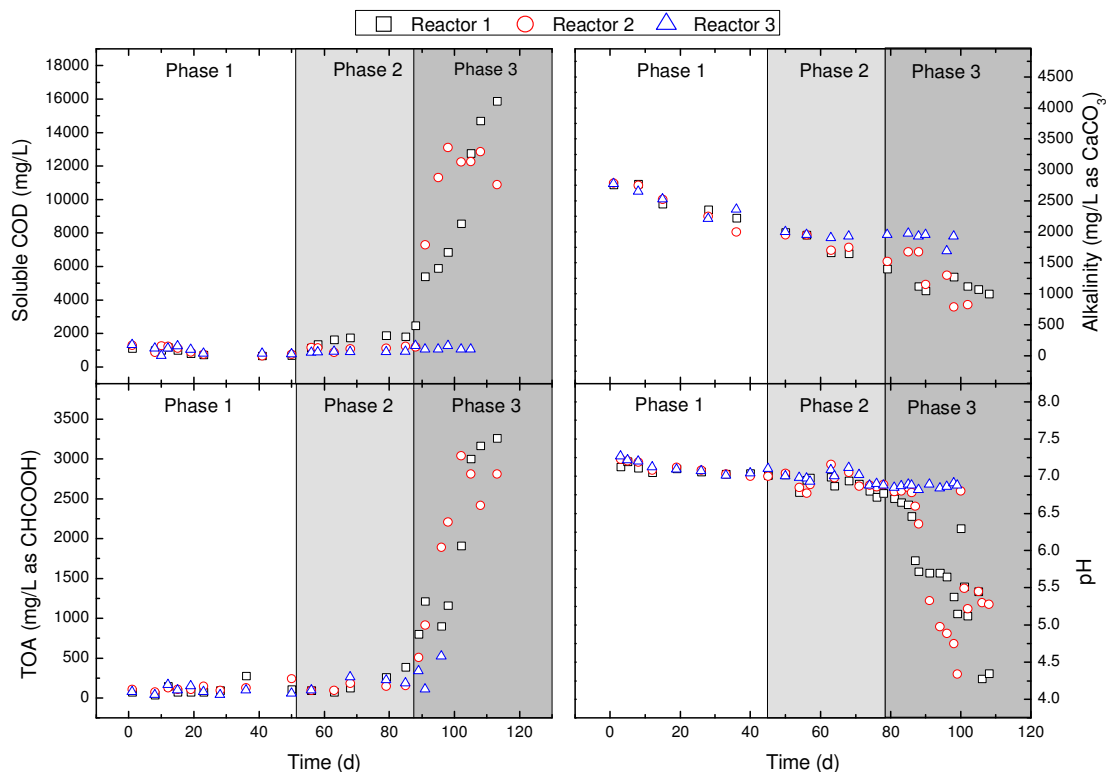


Figure 6: Basic stability parameters: Soluble COD, Alkalinity, TOA and pH (Temperature = 35 ± 1 °C; other experimental conditions are as described in Table 1).

4. Conclusions

Soft drink beverage waste (BW) was evaluated for anaerobic co-digestion with sewage sludge for the first time. Biogas production increase was proportional to the increase in organic loading rate (OLR) from BW addition. The OLR increase of 171% corresponding to 20%(v/v) BW in the feed was the optimum co-digestion ratio, and resulted in an biogas production increase of 191%. Under this optimum condition, co-digestion with BW did not result in any significant impact on digestate quality and biogas composition. The results suggest that sewage sludge can support about 2 kg COD/m³/d OLR increase from a carbon rich co-substrate.

5. Acknowledgement

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